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# AIAA-2001-4958 PHYSICS OF HARD SPHERES EXPERIMENT-2 (PHESE-2)

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## PHYSICS OF HARD SPHERES EXPERIMENT-2 (PH<sup>A</sup>SE-2)

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#### Abstract

The Physics of Hard Spheres Experiment-2 (PHASE-2) seeks to answer fundamental questions about how model atoms change their behavior as more and more neighbors are moved into their neighborhood. The PHASE-2 experiment will use an enhanced microscope to observe colloidal particles crowded in a liquid phase make the transition to a solid phase. In the absence of gravitational masking, we can observe natural phase transitions, and the effects of aiding or frustrating these phase transitions. This allows us to gain an understanding of material behavior that lies at the core of how fundamental material properties manifest in the atomic world. When we increase the number of uniformly sized hard sphere particles in a volume or equivalently look at identical colloidal particles in a large number of sample cells spanning a range of concentrations (volume fractions), we see a fluid go from a liquid phase, to a coexisting phases, to a crystalline phase, and with further crowding on earth to a glassy phase. While dendritic growth is not observed for hard spheres on earth, and the glass transition is, the opposite behavior is seen in experiments in microgravity. We intend to find out why by studying and actively manipulating specially coated particles that behave as hard spheres and, therefore, are only affected by configurational entropy. Choosing a particle size comparable to the wavelength of light permits us to observe model atoms with a microscope. Working in a size range that allows both microscopic and mesoscopic manipulation and control of the samples allows us to probe the essential features of the hard sphere disorderorder transition and the properties of the ordered solid phase that results.

## cientific knowledge to be gained

The information obtained by this experiment will answer questions in condensed matter physics regarding the most fundamental of transitions between liquid and solid phases. An extensive theoretical basis exists, from computer simulations and statistical mechanics theories, as do numerous experimental studies with similar model systems that address one or more aspects of the phenomena. However, gravitational settling complicates the measurements and introduces ambiguities. With the PHASE-2 experiment we seek to sweep aside these ambiguities with a comprehensive study capitalizing on the techniques and knowledge developed in the preceding ground-based, and CDOT and PHASE space shuttle, experiments. The information from this experiment will complement and build upon discoveries made in these previous glovebox and express rack experiments. The history of this microgravity effort and a brief discussion of the results and pleasant surprises that are a result of this work can be found in our sibling paper by Chaikin, et al., entitled "Physics of Colloids in Space Plus (PCS+)," AIAA-2001-5058. For the PHASE-2 experiment we intend to quantify:

- (a) the volume fractions at which dispersions of hard spheres freeze, melt, and become glasses,
- (b) the equilibrium structure associated with each
- (c) the dynamics of Brownian fluctuations in each
- (d) the kinetics of the nucleation and growth of the crystalline phase,
- (e) the response to imposed structural periodicities and deformations.
- (f) the frequency dependent rheology, e.g. elastic constants and viscosity,
- (g) the yield stress and plasticity of the crystalline
- (h) the preference and rate of growth of one phase into another.
- the dendritic growth instability, and
- the effects of polydispersity on nucleation, growth, crystal structure and glass formation.

## The value of this knowledge to the scientific field

The hard sphere phase transition is driven purely by configurational entropy and results from constraints on the packing of impensations the packing of impensations and provides a reference upon which much theory and the part of the pa

computation in condensed matter physics builds. Though extensive, the theory and the database are still incomplete. Further progress requires removing or reducing the experimental constraints imposed by gravitationally induced sedimentation and convection, and extending the experiments over a sufficiently long time scale to accommodate the rather slow dynamics of highly crowded dispersions. The ultimate payoff will be a definitive set of data on this most fundamental of liquid-solid transitions, plus the understanding required to control or modify the structure of densely packed media.

In addition to confirming features quantified on earth and on previous shuttle flights, we expect to elucidate for the first time:

- (a) the controlled growth of annealed crystals through the use of a temperature gradient,
- (b) the stability of the different phases to strong density perturbations and imposed crystal structures,
- (c) the dependence of the rates of nucleation and crystal growth and equilibrium structure on polydispersity,
- (d) the growth of one phase into another, e.g., crystal into glass,
- (e) the evolution of the dynamic structure factor in the glass as it approaches and enters crystallization,
- (f) the local microviscosity and elasticity,
- (g) the equilibrium crystal structure, and
- (h) the dynamics of the dendritic instability.

This data set will permit us to test scalings proposed in the literature, e.g., the relation of the dendritic instability to the critical nucleus size, the temperature density polydispersity relations in the osmotic pressure, the diffusion constants and the nucleation rate in the glass phase.

## The need for working in a reduced gravity environment

To study concentrated systems of particles, we need to match the index of refraction of the particles with that of the solvent. This reduces the amount of multiple scattering and van der Waals interactions to an acceptable level. This is incompatible with density matching, which is needed to avoid significant settling and convection, thus allowing a uniform density at equilibrium. Additionally, the imposition of control fields such as temperature gradients leads to instabilities in any fluid phase in the presence of gravity. Our previous experiments show clear differences between the phase diagram, the crystal structure, and the crystallite morphology between samples measured in 1-g and in

micro-g. These differences are of intrinsic interest on their own, e.g., the facility of crystallization in micro-g of samples which remain in the glass phase in 1-g. The implication is that dynamical processes at volume fractions near close packing are extremely sensitive to gravity.

The main effects of gravity can be seen from the Boltzmann factor governing the particle density for systems as a function  $\exp(-\Delta m g h / k T)$ , where  $\Delta m$  is the buoyant mass. the gravitational samples  $h = kT / \Delta mg$ , is ~ 30 microns. To lowest order this implies a factor of 20 density difference from the bottom to the top of our 100 micron thick cells. To reduce this to an acceptable level (~0.002 variation in concentration) we must reduce the average acceleration to 10^-4 g. (Osmotic pressure effects increase the effectiveness of thermal motion and change the real 7 accompanied requirement to  $\sim 10^{-3}$  g.)

Extensive work in many laboratories, including our own, has attempted to match both the index of refraction and the density of the particles; these attempts have been accompanying by particle swelling and electrical charging. Additionally, work with temperature gradients and multicomponent systems also amplify the fact that working in a microgravity environment enables new and important areas of PHASE-2 needs extended periods of microgravity, since hard sphere colloids have significant density differences between the particle and fluid. Eliminating the driver for sedimentation unmasks the fundamental processes over their natural time scales. Performing these experiments in a reduced gravity environment frees us from the historical constraints imposed by gravity.

## Experimental details for PHASE-2

This section gives an overview of the experimental objectives for PHASE-2. The required measurements build upon CDOT (1) and PHASE (2). They parallel the ensemble average measurements enabled by PCS+ (3) and culminate in a definitive set of measurements of the microscopic and mesoscopic properties of hard spheres, which we refer to in this manuscript as the PHASE-2 experiment (4):

(1) A precursor space shuttle glovebox experiment, the Colloidal Disorder - Order Transition (CDOT) experiment was flown in September 1995 on STS-73. This provided qualitative data similar in concept to that

required in PHASE. Since the hardware was required to be extremely compact to fit into the glovebox (about the size to two shoeboxes), it was limited in scope and capability. However, it provided several spectacular results, namely the observation of dendritic growth instabilities and the crystallization in micro-g of samples that remained in the glass phase on earth.

- (2) PHASE was a fast track space shuttle mission flown on the shortened STS-83 mission and then on STS-94. PHASE provided quantitative data for a select portion of the experiments outlined in this manuscript. There was complete and definitive data on samples for a few concentrations, which served to document the differences between nucleation and growth with and without gravity and provide the basis for further analysis. There was a definitive measurement of the shear elastic constant for much of the phase diagram. To meet the available flight opportunity in 1997 (MSL-1) it was necessary to select a subset of the proposed experiments.
- (3) PCS+ is a reflight with different samples of the PCS mission now aboard Space Station. It differs from PHASE-2 in that it measures ensemble average properties of hard spheres using dynamic light scattering and static light scattering (including Bragg scattering). This experiment is described in detail in an accompanying paper by Chaikin, et al., entitled "Physics of Colloids in Space Plus (PCS+)," AIAA-2001-
- (4) PHASE-2 is a mission that will greatly benefit from the long duration and additional hardware sophistication available in a Fluids Integrated Rack (FIR) supporting the Light Microscopy Module (LMM) planned for Space Station. This experiment is centered on microscopy and allows a sophisticated set of observations and manipulations of equilibrium and non-equilibrium colloids.

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## Nucleation and growth of crystallites

In previous studies performed in PHASE we relied on a combination of low angle light

scattering and observation of the Bragg scattering from the crystallites to analyze nucleation and growth. For a few samples (a few volume fractions) we obtained from the static light scattering probably the best results to date on the start of the formation of crystals, their growth and ripening. However, the low angle measurements were not successful and some of the samples of most interest were not available for study. In particular the coexistence samples that showed dendritic growth and the glassy samples that crystallized in CDOT were not observed. In addition, we have found that polydispersity can be an important variable in the equilibrium properties and hence probably also in the kinetic processes in these samples.

Low angle light scattering is not a part of the microscopy experiment. Rather in PHASE-2 we will directly observe the initial formation of nuclei and follow their growth through static and dynamic 3d confocal microscopy images. The experiment is begun by mix/melting the sample, while it is observed through the microscope. The instrument is then set so that the colloidal particles can be observed in 3d by using confocal microscopy.

Since we have observed dendritic growth instabilities that may lead to non-equilibrium, kinetically formed crystals, it is very useful to study controlled interface growth. In this way we can assure that the system is in equilibrium as it grows or conversely we can study the instabilities. The easiest way to control the growth rate is with temperature

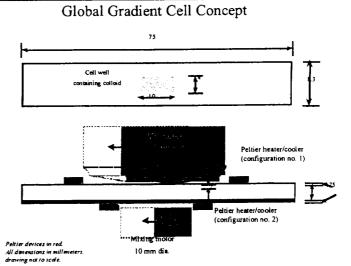
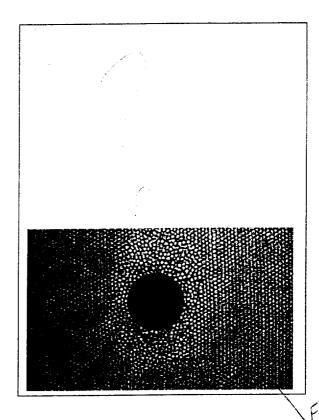


Figure 1. Global gradient cell, which allows a temperature gradient to be used to create a density gradient, which in turn controls the resulting phase diagram.

gradients. In the lab we have taken a sample, which is in the liquid phase, heated one end by 10°C and observed the formation of crystals in the unheated part. In fact this enabled us to grow the largest single hard sphere crystals to date. The amount by which one end is heated controls the "undercooling density" at the other end and hence the growth rate. A somewhat more difficult but much more interesting control would be to create a "hot" region  $(\Delta T \sim 10^{\circ}\text{C})$  approximately 10-100 microns wide across the width of a sample cell which is in the crystalline phase, see Figure 1. This region is depleted of particles and forms a liquid in equilibrium with the crystal on either side. The interface is static. Moving this hot-zone along the length of the sample, melts the solid on the leading edge and reforms it on the trailing edge of this zone. Since the speed of the motion is controllable, the liquid-crystal interface motion is controllable. This process is effectively the same as that used to "zone refine" conventional samples in metallurgy. The process would be studied by microscopy and the crystals measured by confocal microscopy. The onset of the dendritic instability is easily measured by varying the velocity of the zone.

## Rheology

The experiments on PHASE gave the first and definitive results for the shear modulus of hard sphere crystals. The present experiments aim to measure more globally the frequency dependent elasticity, the yield stress, and the viscosity of these systems. The linear regime is probed by monitoring the thermal motion of probe particles that are typically much larger than the particles comprising the hard sphere crystal. This technique is known as microrheology. Basically at short times, or in a purely viscous medium, the probe particle undergoes Brownian motion and from the Einstein relation measures the viscosity of the medium. In an elastic medium the average distance that the probe particle can move thermally is inversely proportional to the shear modulus. This finite motion is responsible for the Debye-Waller factor in conventional scattering. The linear regime experiment is done any time after the samples have equilibrated, typically days after the mix / melt. We want to cover a large frequency / time regime. For long times and displacements 30nm to many microns and times from 1 second to days, the microscope can record the position and motion of the probe particle.



Previously, microrheology has mainly been performed on isotropic samples, either gels, emulsions (etc.) or with small randomly oriented crystallites. The ability to study a single probe particle in a single crystallite by microscopy opens the possibility of observing the anisotropy due to the crystal structure for these hard sphere systems, thus reflecting their phonon spectrum.

For nonlinear rheology the optical tweezers are used in conjunction with the probe particles. The tweezers can grab a probe particle and move it either at a steady velocity or at a fixed oscillation frequency. As shown in Figure 2, the microscope observes the particle displacement. The distance from the particle to the center of the trap gives the drive force. From the force and motion both the linear and nonlinear viscoelasticity can be obtained over limited ranges typically 1-100 pN and 30-0.03 Hz. Properties such as the yield stress can be obtained from the nonlinear rheology.

Imposed patterns, susceptibilities and metastable states

The basic idea of this set of experiments is to measure the susceptibility of the hard sphere system to form different equilibrium and non-equilibrium structures, to measure the stability of these structures and to see whether different imposed structures can be grown, controlling the final solid. The susceptibility experiment is performed in the liquid state samples. Either optical tweezers, the electric field gradients from a lithographic grid on the sample wall, or indentations in the sample cell cover slips induce in the colloidal layer next to the surface a specific two-dimensional arrangement. The former two are preferable in so far as the strength of the imposed pattern can be varied and, in particular, turned off. Microscopy and confocal microscopy are then used to observe how far the induced structure propagates into the bulk sample. For example, the pattern can correspond to the close packed face (111) of a close packed crystal, the (200) face of fcc (which forces fcc), the (1100) face of hcp (which forces hcp), or the (110) face of bcc. Examples of how easy it is when working with laser tweezers to form a pattern out of 0.92, diameter particles is shown in Figures 3 and 4. Figure 4 actually is a still from a movie where we show the first letter "A" in the word "NASA" being launched like a space shuttle under the control of laser tweezers. This demonstrates that we can selectively manipulate all or select portions of patterns formed



Figure 3. Princeton logo dynamically formed with laser tweezers from 0.92. diameter particles. This entire image is the width of a human hair.

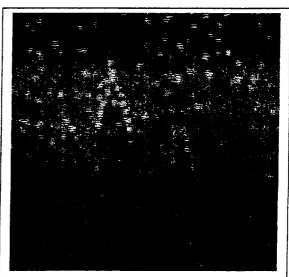
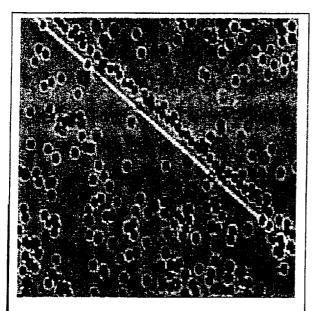


Figure 4. Snapshot from a movie created using laser tweezers. The movie shows the letter "A" in "NASA" launching like a space shuttle. This demonstrates that we can use laser tweezers to selectively manipulate some or all of the patterns they create from colloidal particles.

from colloidal particles with laser tweezers. Figure 5 shows snapshots from a movie displaying two elements of an electric field grid being turned on. The intensity of the electric field gradient is adjustable and additional elements can easily be added for growing 3-d structures from just such a substrate. The spheres in the movies are 2.13 micron diameter polystyrene particles, diluted in deionized water. The gap between the electrodes is ~1 micron wide, the voltages are in the range 100 mV-1 V rms, this gives a field strength ~1 V/micron. The jumping is achieved by switching the frequency over the resonance (at about 700 kHz). At high frequencies (MHz) the spheres are repelled, at lower (~100 kHz) they are attracted. (And at ~10kHz you get rolls (AC electrophoresis), you can see just a little of this at the end of the movie.)

A similar set of experiments can be done with crystalline samples, but in two ways. For the first way, the samples are melted by mixing, the imposed surface pattern is turned on, and the sample nucleates and grows from this seed. Microscopy and local confocal microscopy are used to see the range of the imposed pattern. As demonstrated by van Blaaderen, there is the chance to grow large oriented single crystals by this technique. The ideal experiment of this category is to take a sample in the liquid phase, impose the pattern and start nucleation, and then warm the far side of the sample to control the growth of the nucleated crystal. It



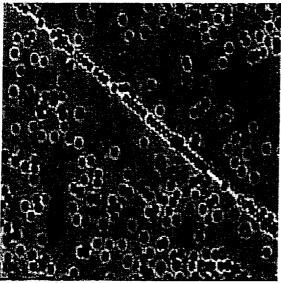


Figure 5. Snapshots from a movie displaying two elements of an electric field grid being turned on in the presence of 2.13 diameter particles. The intensity of the electric field gradient is adjustable and additional elements can easily be added for growing 3-d structures can from such a substrate.

will be most interesting to see whether large crystals of a non-equilibrium phase (e.g. bcc) can be induced. In the second set of experiments the imposed patterns are turned on after the crystals have grown. Depending on the strength of the forcing pattern, we will microscopically observe strain fields or the nucleation of a different structure, which tests the stability of the bulk equilibrium structure.

In the glass phase the patterns will be imposed at various times after mix/melt to see whether the nucleation of the crystal is hindered after a glass-like phase sets in. From our previous shuttle experiments, and recent ground-based experiments by P. Segre and D. Weitz, we know that in low-gravity the glass phase does not exist. This study will aim to understand why it may exist in 1-g.

As another part of this study we plan to use the power from the optical tweezers to locally heat a region in a solid sample to melt it and observe the recrystallization. Of special interest is to see whether there is any difference in behavior as the volume fraction is varied through the "glass" transition concentration.

## Glass transition, polydispersity effects

Ground-based experiments have shown that samples in the volume fraction range 0.58 to 0.63 remain uncrystallized in a rigid glass-like form for periods of weeks to years. Similar samples in low gravity crystallize readily. Ground-based DLS experiments have shown that these samples have a characteristic long time behavior and a self-diffusion constant that goes toward zero as the "glass" transition is approached. The important experiments tend to be in the metastable liquid phase before the crystal forms (at volume fractions below 0.58) and then in the glass phase. In some cases the liquid and glass phases are preserved by the polydisperse nature of the samples. We will investigate both the pre-crystallization dynamics and the effects of polydispersity. This experiment will benefit from having hundreds of small sample cell (see Figure 6), which will allow us to incrementally span a full range of sample concentrations.

Directly after high volume fraction samples are mix / melted we will use a time history created with confocal microscopy, confocal microscopy with tracer particles, and microscopic particle tracking to evaluate the collective and self-diffusion of the same samples in 1-g and micro-g. The samples to be studied will also use different polydispersities from 1 to 5% in radius. We also plan to have some samples of different polydispersity for study at lower volume fraction to test whether the polydispersity effects nucleation, growth and especially "equilibrium" crystal structure. Although it is generally agreed that the structure should be FCC, experiments show mostly RHCP. The polydispersity may kinetically stabilize the more random structure.

#### Experimental procedures

where's figure

The experiment will involve making a series of measurements on approximately 200 different samples of different concentrations (i.e. volume fractions of PMMA in an index matching fluid). The samples will be made from a mixture of decalin, tetralin, and PMMA spheres with a stabilizing polymer grafted onto them. The decalin (index = 1.48) and the tetralin (index = 1.54) will be mixed in a combination which index matches the swollen PMMA (index = 1.51). This will address multiple scattering concerns which will aid in probing the particle dynamics at different concentrations. In addition, some samples will contain probe particles, which differ from the above particles in having different size, index of refraction or containing fluorescent dyes.

The PHASE-2 sample volume fractions (PMMA spheres suspended in decalin / tetralin) will vary from 0.45 to 0.63 in increments of approximately 0.003.

Once in orbit, the particular experiment will be initialized by translating the sample to the observation position and rotating the objective turret to the mix / melt position. After melting, each sample will be examined by the microscope and by static confocal microscopy to ensure that complete melting has occurred. Then the particular experimental sequence begins. For nucleation and growth experiments the observations begin immediately. For other experiments the sample is moved to another position for After equilibration the sample is equilibration. translated back to the experimental position in front of the objective and the observations are made and/or the system is perturbed by laser tweezers, temperature gradients and/or imposed patterns and observations are made through the microscope ûse the imaging techniques.

#### Summary

This paper provides a general description of the science objectives for PHASE-2 and how they are being accomplished.

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